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Near Net-Shape, Ultra-High Melting, Recession-Resistant Rocket Nozzles II: Low Cost Carbon-Carbon Technology for use in Ultra-High Temperature Oxidative Environments

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Carbon-carbon composites are ideal materials for high temperature structural uses, such as in rocket propulsion components, hypersonic vehicles, and aircraft brakes. In spite of their excellent properties, the use of carbon-carbon composites has been limited because of their high cost and rapid oxidation at elevated temperatures. Two novel approaches to solving these problems are described and these approaches are employed along with a ZrC/W-based nozzle insert to fabricate and test a recession-resistant carbon-carbon rocket nozzle as a potential replacement for solid tungsten nozzles.

1. Introduction

Carbon-carbon (C-C) composites, which consist of carbon fibers in a carbon matrix, [1-2] possess a unique set of properties that make them ideal materials for high temperature structural uses, such as in rocket propulsion components, hypersonic vehicles, and aircraft brakes. These composites are stronger and stiffer than steel, while less dense than aluminum. In addition, they not only maintain their mechanical properties to temperatures in excess of 3000° C, but the composites' material properties actually improve with heating as the non-ordered carbon is graphitized. In spite of their excellent properties, the use of carbon-carbon composites has been limited because of their high cost and rapid oxidation at elevated temperatures. Extensive efforts over the last 30 years have been unable to resolve these two issues. Now, however, new technical approaches to deal with these two issues have been developed. These approaches will be addressed in this communication along with a demonstration of the technologies developed. The demonstration involves a test firing of a rocket nozzle fabricated utilizing a marriage of the carbon-carbon technologies with the displasive compensation of porosity (DCP) process [3-4], to produce a much lighter weight replacement for solid tungsten nozzles currently used in operational systems.

1.1 Fabrication of Carbon-Carbon Composites

The high cost of C-C composites is principally due to the costs associated with the amount of time needed to fabricate the composite which involves the forming and densification of the fiber preform. Except in the case of very high performance or high thermal conductivity fibers, the cost of the raw materials is a very small part of the final composite cost. Likewise, in most applications, the cost associated with forming a preform by the placement of the fibers in the orientations desired to carry any structural load is also not the main cost driver. In the fabrication of most of C-C composites, the major cost is due to the densification of the preform in which a hydrocarbon

precursor to the carbon matrix material is placed in the voids between the fibers and the fiber bundles and then thermally converted to a carbon or graphitic matrix. This is a very expensive process that can take up to nine months per part.

Although there are many different hydrocarbon matrix precursors, there are just two major paths for densifying a carbon fiber preform with these precursors. One process involves infiltrating the composite preform with a hydrocarbon gas, such as methane [5] or propylene [6], while the other process involves impregnating the fiber preform with a liquid hydrocarbon such as a petroleum pitch [7], coal tar pitch[8], refined pitch [9-10], mesophase pitch [11-13], or an organic resin such as phenolic [14]. In all cases the densification process is followed by carbonization in which the matrix precursor is pyrolyzed inside the preform converting it to carbon. In most manufacturing processes, the carbonization step is followed by graphitization in which the partially densified preform is heated to temperatures in the range of $2000°$ C to $2800°$ C. This heat-treatment not only converts some types of carbon fibers and matrix carbons to a graphitic structure, enhancing both thermal conductivity and mechanical properties [15], but also opens up a crack network [16] that enhances the effectiveness of further densification steps.

There are numerous densification cycles in most carbon-carbon composite fabrication processes that are necessitated by both the char yield of the matrix precursor and the inhomogeneous densification of the composite. That is, since the carbonization process removes non-carbon atoms from the matrix precursor, the char yield, which is defined as the ratio of the amount of carbon remaining to the amount of hydrocarbon matrix precursor, must be less than 100% and is usually between 40% and 85%. In addition, gases formed during the pyrolysis process tend to push some liquid species out of the fiber preform. As a result of these two processes, void space remains in the

partially densified preform after each cycle. This residual void space must be densified on subsequent cycles, which means that ultimate composite density is approached asymptotically.

The inhomogeneous densification of the composite is manifested in two ways. The predominant effect is the density gradient that exists because both gas phase infiltration and conventional liquid phase impregnantions preferentially densify the exterior of the preform. A secondary concurrent effect arises from the closing off of small porosity during deposition. In gas phase infiltration, the gradient results when the gaseous precursor impinges on the hot preform surface and pyrolyzes before it can diffuse very far into the preform. There are several approaches to solve this problem of preferential deposition on the exterior of the preform. These included adjusting residence time of the precursor in the fiber preform [17], employing thermal gradients [18-19], or utilizing the forced flow of matrix precursor gas [20]. Despite these various process modifications, uniform gas phase densification is usually limited to composites with the smallest dimension less than 5 cm.

In a similar manner, a densification gradient results from liquid phase impregnation processes because it is difficult to get the liquid impregnant into the central portion of the preform. This has been assumed by the industry for years to be due to the high viscosity of the precursor but will be shown in this work to be due principally to the lack of wettability of the precursor. To solve this problem, the industry has employed solvents to decrease the viscosity and high pressure to force the impregnant into the preform. Even with this costly process, however, a significant gradient still exists unless the part is subjected to more than ten densification cycles, which is usually cost prohibitive.

As a consequence of the density gradient, after the composite is graphitized, all current commercial densification processes require that the outside of the partially densified composite be

machined to remove material. This is necessary because the preferential densification of the exterior of the preform blocks the surface pores so that additional densification cannot occur. Because of the shortcomings of current densification processes, the infiltration/impregnation-carbonization-graphitization-machining cycle must be repeated up to 10 times depending on the final density required for the application. Since each cycle requires 3-4 weeks, the densification process is very long and costly.

By understanding and addressing the issues associated with the shortcomings of current commercial processing, the In-Situ Densification Process, developed at the Propulsion Directorate of the Air Force Research Laboratory (AFRL) and described below, is able to avoid inhomogeneous densification and reduce the time required from many months to less than two weeks, with an associated dramatic decrease in cost.

1.2 Elevated Temperature Oxidation of Carbon-Carbon Composites

The principle use of carbon-carbon composites has been and continues to be in the realm of high and ultra high temperature applications such as aircraft brakes and furnace elements as well as in leading edges of hypersonic vehicles, rocket nozzles, exit cones and nose tips. At temperatures above 500 °C, carbon oxidizes rapidly and therefore must be protected from oxygen for maximum performance above this temperature. However, in many applications such as rocket nozzles and nose tips, carbon is unprotected and therefore oxidizes. This oxidation in rocket nozzles results in an enlarged throat region, which leads to a subsequent reduction in thrust, range, and payload for systems utilizing carbon-carbon composite throats. This reduction in performance increases as the throat diameter decreases, and therefore is most severe for tactical missile nozzles.

Many C-C components in use are currently unprotected because the oxidation protection of carbon-carbon composites at ultra high temperature (>2400 °C), or over a wide temperature range,

has proven to be an impossible problem to solve over the last 25 years. The main impediment is that carbon-carbon has a thermal expansion coefficient of nearly zero. Thus, any coating that is applied to the surface will have a thermal expansion coefficient 3 to 20 times as great. During thermal cycling and at high temperatures, a lot of stress develops due to the thermal coefficient mismatch between the composite and the coating. This thermal stress causes the coating to crack and spall off.

Numerous approaches have been used over the years to try to solve this problem. These include graded coatings [21], layered coatings [22], as well as impregnations [23] and functionally-graded coatings [21]. Perhaps the best-known approach is that used for the leading wing edges on the Space Shuttle. This is a silicon carbide coating for oxidation protection and a silica over-layer for crack sealing. Thus, when the leading edge heats up, the silica will melt and form a glass to fill the stress cracks in the silicon carbide. This system works well on the Space Shuttle but does not work at lower temperatures or at temperatures higher than 1600 °C. Thus, an oxidation protection coating for carbon does not exist in the temperature regime in which rocket nozzles and nose tips operate.

Because conventional processing involves graphitization temperatures as high as 2400 °C, it is not possible to place an oxidation protection coating on the composite surface before processing is complete because it will not survive intact at these high temperatures. One work-around for this problem is that coatings are placed on the composites after processing. However, the disadvantage of this technique is that it is not possible to maximize the bond between the coating and the surface of the composite. That leaves the less-than-desirable choices of placing a thick coating on the surface, with the risk of having it crack and possibly spall off, or placing a thin coating on the surface, which will not survive in an erosive environment. These problems associated with the

oxidation protection of carbon-carbon composites are solved by employing the Inverse Processing approach described below.

2. Unique Approaches to Solve Intractable Problems

The Propulsion Directorate of the Air Force Research Laboratory (AFRL) at Edwards CA has come up with solutions to both the cost and oxidation problems associated with carbon-carbon composites. AFRL's In-Situ Densification Process addresses the issue of the high cost associated with these composites, while the Inverse Process addresses the problem of oxidation.

2.1 In Situ Densification

In Situ Densification is an impregnation process that, in contrast to commercial processes, is able to rapidly densify the composite uniformly because it addresses the conflicting requirements of the impregnant's low viscosity and good wettability on the one hand and the need for a high char yield carbon precursor on the other.

The key to the success of this process is two-fold. First, it was realized that it is not just the viscosity of the impregnant that is important, as had been assumed for 30 years, but also the contact angle of the impregnant on the surface of the preform. Thus, it has been necessary to employ high pressure in commercial processing for many years, mainly because the impregnants do not wet the surface of the preforms. Viscosity is actually a secondary factor. The second insight was that good wettability, low viscosity, and high char yield do not exist in the same molecule. That is, low molecular weight molecules exhibit lower viscosity and better wettability, while high char yield requires much larger molecules.

To produce a high quality carbon matrix these insights were combined with the results of the extensive work in the production of mesophase pitch by Professor Mochida. [24-26]. Mesophase

pitch is the liquid crystal precursor to high quality graphite and for carbon matrices has been produced for more than 30 years by the thermal treatment of coal tar or petroleum pitch. Professor Mochida's important contribution was to realize that mesophase pitch can be produced by the polymerization of pure polyaromatic feedstocks, such as naphthalene [27]. Mesophase is commercially produced by this process by Mitsubishi Oil [28] and is used principally in the manufacture of carbon fibers [29-30]. Mitshibutsi Oil and others [31] have used this pitch as a matrix precursor for carbon-carbon composites. Although this pitch produces a high quality matrix material with a char yield of ~ 84%, the process still suffers from the many disadvantages mentioned previously for other processes. In addition, this pitch needs to be oxidatively stabilized to keep it from melting at high process temperatures.

The application of Professor Mochida's approach in the In Situ Process is to use the monomer precursor to the mesophase pitch rather than the pitch itself as the impregnant. Thus, a low viscosity material such as naphthalene, which totally wets carbon surfaces, will enter a carbon fiber preform as easily as water enters a sponge. This not only results in a uniform density, but, in addition, there is also no need for machining after densification to open up the surface pores. After the impregnant has filled the preform, in the second step of the process it is polymerized into a carbon matrix precursor that has a high carbon yield, which means that fewer cycles are needed to bring the composite to final density. The carbon matrix precursor is then pyrolyzed to produce a high quality carbon matrix.

Not only does the In Situ Process produce a uniformly densified composite from any type of fiber preform (woven, braided, 1-D to n-D, felt, etc.) but, in contrast to other processes, there does not appear to be a size limitation. That is, preforms with diameters up to 45 cm and lengths to 183 cm have been uniformly densified with a gradient of ~ 2%. In addition, another unique feature of

this technology is the ability to join carbon-carbon parts together with a seamless joint that possesses significant mechanical strength compared to the original parts.

Finally, it should be noted that, in contrast to other processes, it is not necessary to graphitize the composites, which results in significant time and energy savings. Even without graphitization, the ablation and erosion performance of the In-Situ material equals that of commercial material when exposed to the exhaust of either liquid rocket engines or solid rocket motors. Thus, in most applications, it is not necessary to graphitize the material. However, when this material is exposed to graphitization temperatures in excess of 2200° C, a highly graphitic material with high thermal conductivity results. The significance of this result is that it is possible to use the low-cost liquid phase process to produce a C-C composite with the properties of a composite produced by employing a gas-phase process.

2.2 Inverse Process

Unlike current densification technologies, the In-Situ process is a relatively low temperature process. That is, since the properties of the carbonized preform are so good and the pore structure remains open, there is no need for graphitization, and thus the composite is only exposed to processing temperatures up to 1000° C rather than 2400° C. With this lower processing temperature, an alternative approach to the oxidation protection of carbon-carbon is possible. With In-Situ processing it is possible to construct a thin shell or an insert out of the oxidation protection material, maximize its properties, and then build the carbon-carbon composite around the insert. This approach, so-called inverse processing, has worked flawlessly. Carbon-carbon rocket nozzles fabricated using inverse processing containing both metallic and ceramic inserts have performed exceedingly well.

In practice, the thin free-standing metallic or ceramic insert is fabricated by any one of a number of processes, such as casting, molding, isostatic pressing, plasma spraying, electro-deposition, etc. The properties of the insert are then enhanced using the appropriate process such as sintering, annealing, densification, etc. A compliant layer is placed on the exterior of the insert and then a carbon fiber preform is wound, laid-up, woven or braided around the insert. The fiber preform is then densified to the appropriate density and subsequently machined to the dimensions required for the rocket motor.

3.0 Experimental

A ZrC/W-based nozzle insert was manufactured by MetaMateria Partners LLC of Columbus OH using the displacive compensation of porosity (DCP) process [4]. The completed insert was delivered to the Propulsion Directorate of the Air Force Research Laboratory at Edwards CA, where it was prepared for firing in the Pi-k demonstration motor. This preparation consisted of wrapping the insert with continuous T-300 carbon fibers, and densifying the fiber preform using the In Situ Densification process [32] with naphthalene as the matrix precursor. The carbon-carbon composite surrounding the insert was then machined to the final shape required by the test motor. A restraining collar, which actually holds the nozzle in the motor, was then bonded to the carbon-carbon composite that surrounded the exit region of the nozzle.

4.0 Results and Discussion

Tungsten is used as a rocket nozzle material in several Air Force and Navy operational systems. Although it performs adequately, it has a very high density (19.3 g/cm^3). Thus, there is a significant payoff to reduce the weight associated with these nozzles, if possible, to also reduce the recession rate of the throat region associated with the harsh environment to which these nozzles are exposed. The combination of the displacive compensation of porosity (DCP) process described

elsewhere [3] and the inverse process described above have the potential to drastically reduce the weight of a nozzle that can be used to replace a solid tungsten nozzle.

The material used in this experiment to make a nozzle insert by the DCP process was a composite of zirconium carbide and tungsten. Because ZrC has a significantly lower density than tungsten (6.63 vs. 19.3 g/cm^3), the DCP-derived ZrC/W-based composite has the potential to reduce the weight of a solid tungsten nozzle by 10-57%. For example, the density of a 20 vol% ZrC composite is 10.1% lower than that of pure tungsten; the density of a 50 vol% ZrC composite is 40.6% lower than pure tungsten; and the density of an 80 vol% ZrC composite is 56.8% lower than that of pure tungsten.

There is the possibility of very significant additional weight reduction if a thin insert of this metallo-ceramic composite material is used in place of a monolithic metallo-ceramic composite nozzle. For example, if a carbon-carbon composite material with a density of ~1.60 g/cc is used as a structural backing material to support the insert, the total weight of the nozzle will be only a fraction of that of a nozzle composed entirely of metallo-ceramic composite material. The resulting macro-composite structure composed of two micro-composite materials is tailorable and thus could possess a wide range of properties.

The ZrC/W-based insert (Figure 1) with a throat diameter of 1.10 cm was delivered by MetaMateria Partners LLC of Columbus OH [4] to AFRL, where it was backed with a carbon-carbon composite. The C-C composite that surrounded the insert had two functions in addition to reducing the overall weight of the completed nozzle: (1) to bring the insert to the final external dimensions required for the motor, and (2) to provide mechanical backing to the insert while the interior of the insert was exposed to the high gas pressure during the firing.

The C-C backing was machined (Figure 2) and a retaining ring was bonded to the carbon-carbon backing. The completed nozzle was then placed in a Pi-K test motor (Figure 3), which had been previously loaded with a solid propellant having a 19% aluminum loading. The temperature that the throat experienced upon combustion of this propellant was predicted by thermal-chemical calculations to be 3245 K.

The propellant grain was ignited and burned (Figure 4) for 4.6 seconds with a maximum pressure of 3.6 MPa. The maximum pressure was limited by the grain design and the amount of propellant that was available for the test. During the test, the nozzle performed extremely well, as seen in the pressure trace (Figure 5). The nozzle insert survived the critical initial thermal shock of heat-up and gave a very smooth pressure trace until the small perturbation at the end of the trace. This blip in the pressure was due to the formation of axial hairline compression cracks in the entrance of the ZrC/W insert. These cracks resulted from the fact that there was not a sufficient compliant layer thickness between the insert that was expanding as it was heated and the C-C backing that did not change dimension during the firing.

In designing and manufacturing a C-C backing for the insert, one must allow enough space for expansion but not so much that the insert is unsupported or is loose. Previous firings with other much thinner ceramic and metallic inserts did not show these hairline cracks. However, this was the first test with an insert of this composition and thickness. Since this is a new material, a much thicker insert than has been tested previously, and the ultra high temperature thermal expansion behavior was not known in advance, adequate space for expansion was not provided.

Upon post examination of the nozzle insert, it was observed that the throat had remained intact and the dimensions of the throat had not changed, except for some alumina deposition that can be seen on the exit of the insert (Figure 6). In addition, the surface of the ZrC/W looked

unaffected by the firing with no roughening or recession observable. The data obtained during the firing and the post analysis indicated that the nozzle insert performed very well. It was also concluded that the insert would have survived a much longer test since the hairline cracks in the entrance did not allow exhaust gases to reach the carbon-carbon backing.

Conclusion

A recession-resistant rocket nozzle that utilized a ZrC/W-based insert that was backed with a low cost carbon-carbon composite was tested in a solid rocket test motor. The ZrC/W- based insert fabricated using the displacive compensation of porosity method performed very well with no apparent recession.

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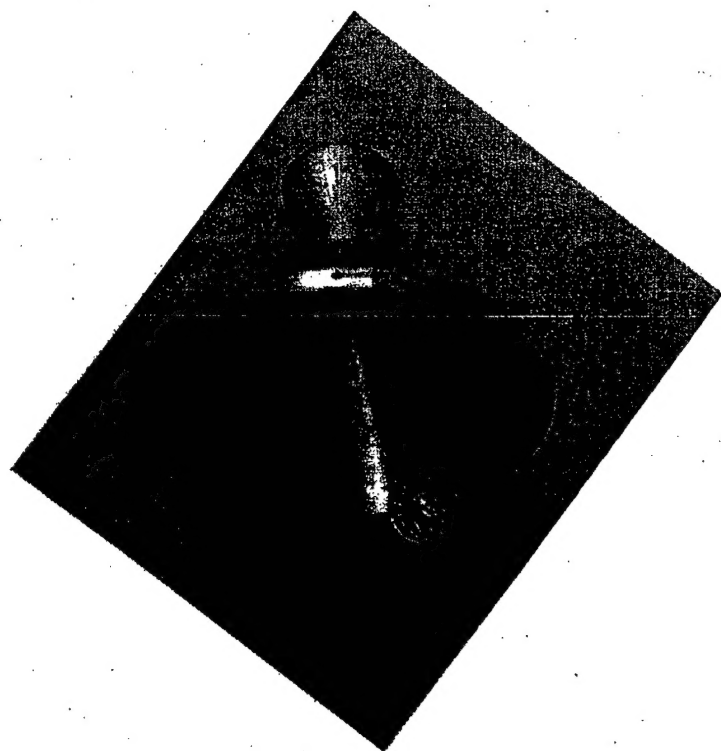32. P.G. WAPNER, W.P. HOFFMAN, J.P. JONES, U.S. Pat. No. 6,309,703
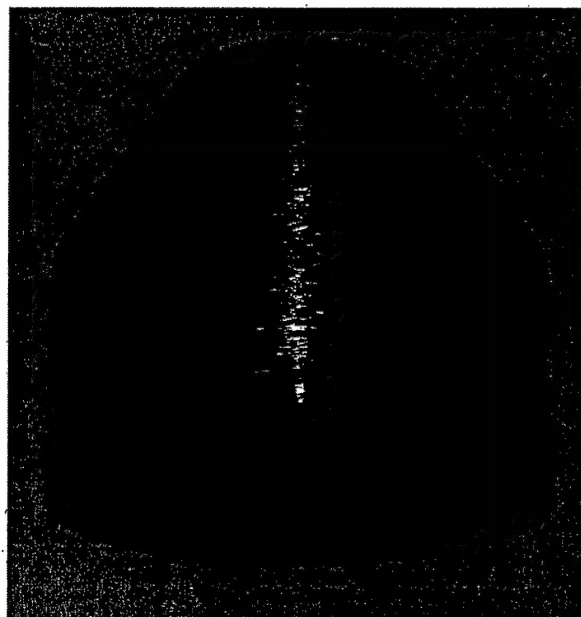
Figure 1 ZrC/W nozzle insert as-received

Figure 2. ZrC/W nozzle insert after it has been wrapped with carbon fibers, densified, and machined to size for the test motor
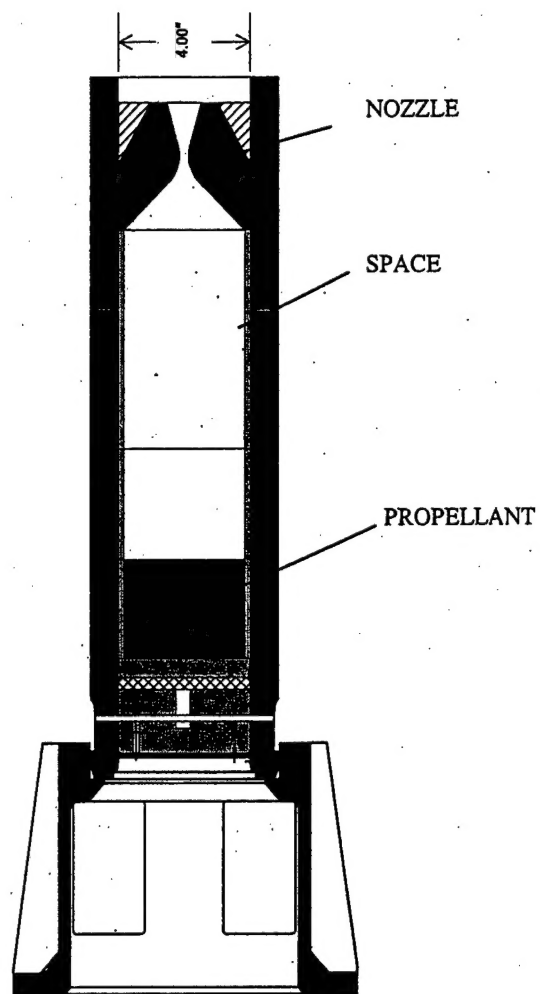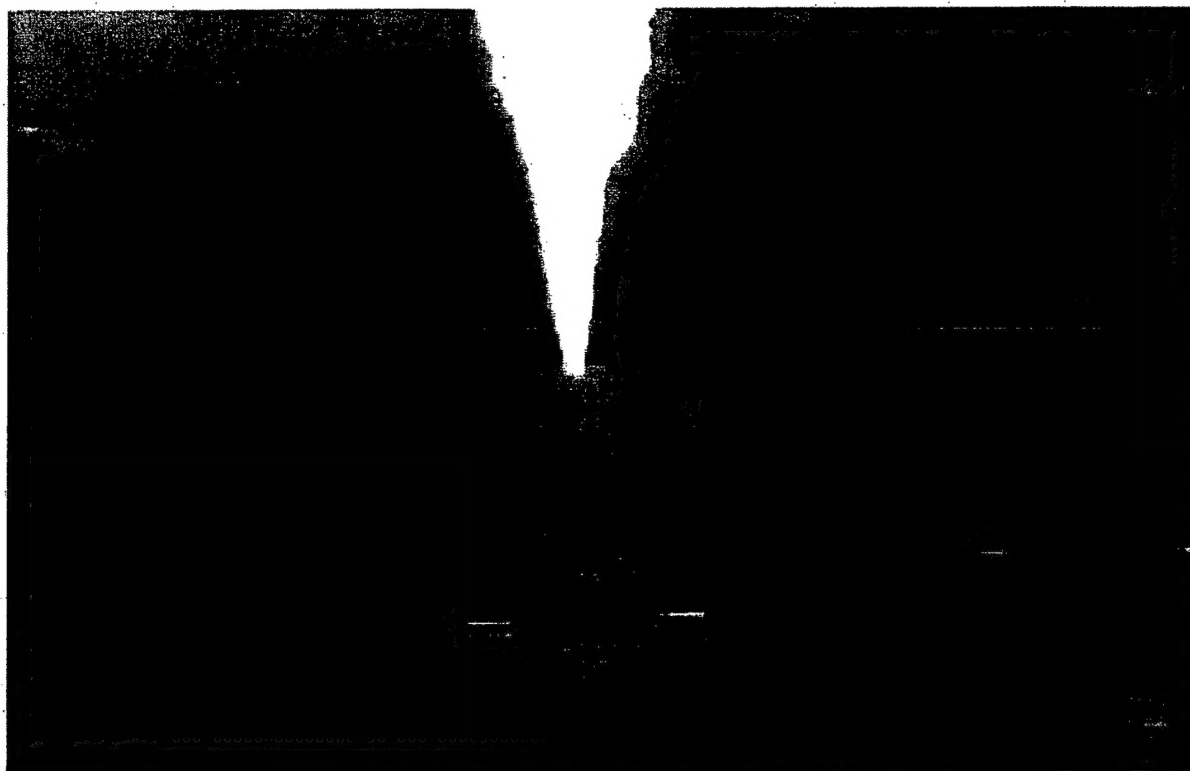
Figure 3. Schematic of Pi-K test motor

Figure 4. Test Firing of Pi-K motor with carbon-carbon over-wrapped ZrC/W insert
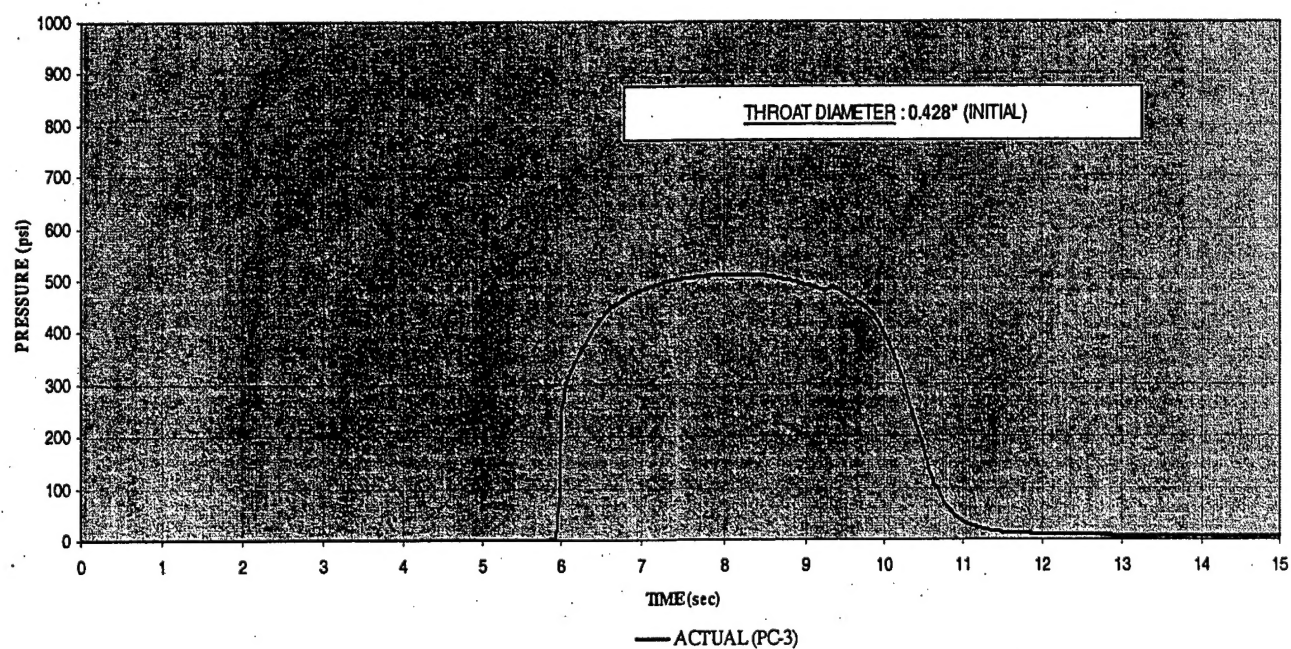
W-ZrC-NOZ-001 PRESSURE vs TIME

THROAT DIAMETER : 0.428" (INITIAL)

ACTUAL (PC-3)

Figure 5. Pressure trace during test firing.

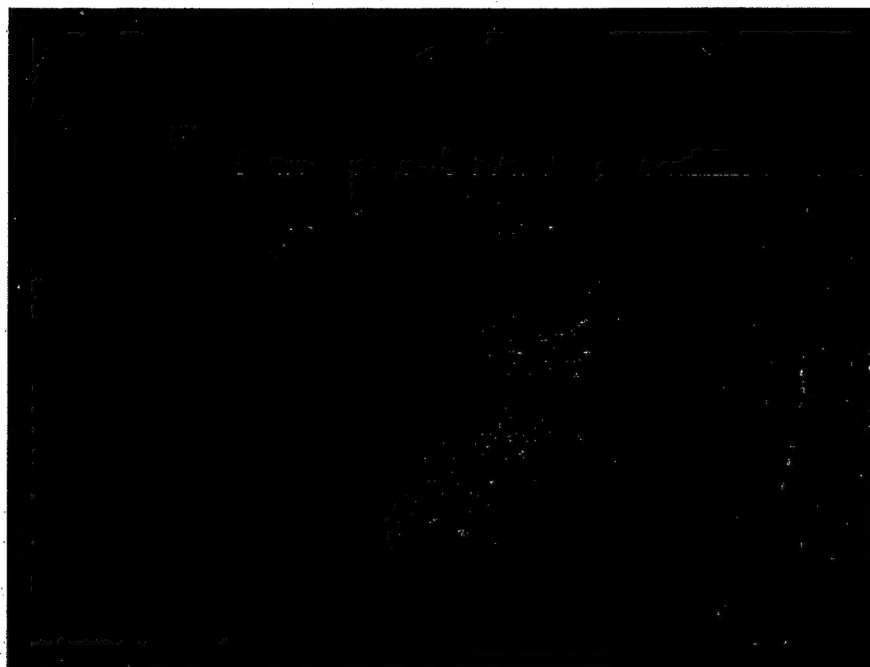Approved for public release; distribution unlimited

Figure 6. Exit of nozzle showing aluminum oxide deposit on surface of ZrC/W insert that is surrounded by the carbon-carbon composite.